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1954

TBP PROCESS

PILOT PLANT DESIGN REPORT

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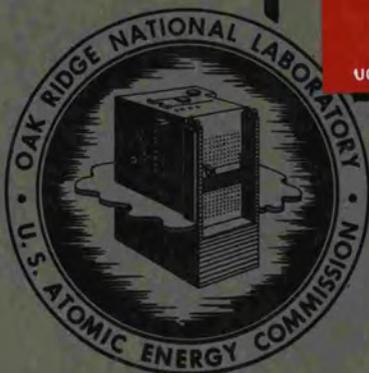
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TECHNICAL DIVISION

CHEMICAL PROCESS DESIGN GROUP

TBP PROCESS

PILOT PLANT DESIGN REPORT

BY

R. P. Milford

Date Issued

JAN 07 1956

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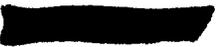
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## 0.0 Abstract

A set of stainless steel columns and accompanying process equipment, including a product evaporator, was installed in Bldg. 706-HB to demonstrate the TBP process for recovering uranium metal waste. Tributyl phosphate, diluted with Varsol, is used as an extractant in one cycle of continuous liquid-liquid countercurrent extraction, scrubbing, and stripping.

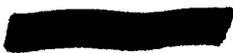
## 1.0 Justification

A solvent extraction process has been developed for the recovery of uranium from metal waste. The procedure involves the solution of waste sludge in nitric acid to a free acidity of about 3 M, extraction with a 15% tributyl phosphate-85% Varsol mixture, and subsequent stripping of uranium from the solvent with water. In the laboratory uranium from both ORNL waste sludge and Hanford waste supernate was recovered in one cycle of batch counter-current solvent extraction with a loss of 0.1%, and a beta decontamination factor of greater than  $10^4$ . Further work was done on 1 in. laboratory columns to confirm the uranium losses and decontamination factors. Early laboratory work gave such encouraging results that it was decided to make a pilot plant installation to demonstrate the feasibility of this process as a method of recovering both Hanford and ORNL metal wastes.



## 2.0 General Description of Process

In preliminary discussions laboratory personnel outlined the following conditions to be used as a basis for design. (1) Feed preparation was to be performed in an adjacent cell and was not to be considered part of this installation. From the feed preparation area the uranium bearing solution would be transferred to an adjustment tank where it would be made 70 to 120 g./l. in uranium, and 3 N<sub>2</sub> in nitric acid. (2) The scrub was to be 3 N<sub>2</sub> nitric acid. (3) The extractant was to be a mixture of 15% tributyl phosphate and 85% Varsol. (4) The stripping agent was to be demineralized water. (5) A column packed section height of 18 ft consisting of 10 ft of extraction and 8 ft of scrub was estimated to be the minimum requirement. The strip column was to be as high as possible and still maintain cascade flow conditions. (6) Flow volume ratios of 6 organic, 2 feed, 1 scrub with a 6-6 organic to strip ratio were suggested. It appeared that a column constructed of 1½ in. IPS pipe would give a 40 hr run using a total throughput rate of 300 gal/hr/sq ft at less than 50% of flooding. (7) The product was to be evaporated to a solution 2M in uranium.



3.0 Design Data

3.1 Equipment Volumes

Based on the design data above, the size of the feed tank has been calculated as follows using 25% tank freeboard and 0.014 sq ft as the cross sectional area of 1½ in. Sch. 40 pipe:

$$\text{Volume} = (300)(2/9)(40)(0.01414)(1.25) = \underline{47.1} \text{ gals. or } 178.4 \text{ l.}$$

The volume of the scrub feed tank is one-half that of the feed tank or approximately 25 gal., and the volume of the solvent feed tank is three times that of the aqueous feed tank, or 150 gal. The strip feed tank is also 150 gal. Since the strip is only demineralized water, the tank may be filled under pressure during a run and a smaller tank is adequate. A 150 gal catch tank is required for both the product and the waste solvent.

3.2 Column Dimensions and Pressure Pot Locations

Although 8 and 10 feet have been set as the minimum packed section lengths for the scrub and extraction sections respectively, it is desired that the column be built as long as available head room will permit. The cells in Bldg. 706-HB have 29½ ft of head room available. Allowing 2½ ft total free space above and below the extraction column, taking into account the length of the various spool pieces, and proportioning the remainder of the column on an 8 to 10 ratio, a scrub section length of 9 ft-10in. and an extraction section of 12 ft-6 in. results.

Column Dimensions and Pressure Pot Locations (cont.)

To determine the optimum height of the pressure pot, for the extraction column it is necessary to calculate the hydrostatic conditions prevailing when the interface requires the least amount of instrument air pressure and those prevailing when the highest amount of air pressure is required. These conditions are met respectively when the column is running in equilibrium at the maximum flow rates, and the opposite extreme which is nearly attained at start-up when the scrub section contains scrub solution, the extraction section contains feed solution, and the line to the pressure pot contains scrub feed solution.

To find the minimum height of the pressure pot for the extraction column, the total head in the column is set equal to the total head on the jackleg. The total head in the column is made up of the following terms:

- (a) Solvent above the interface
- (b) Solvent flowing through aqueous phase
- (c) Aqueous in scrub section
- (d) Aqueous between solvent feed point and aqueous discharge.

The total head on the discharge line is as follows:

- (f) Aqueous in discharge line
- (g) Air pressure on pressure pot.

Column Dimensions and Pressure Pot Locations (cont.)

The items listed below are the basic information necessary to perform the calculations:

Sp <sub>gr</sub> of 3N HNO <sub>3</sub> scrub		1.1
Sp <sub>gr</sub> of solvent mixture		0.78
Maximum feed sp <sub>gr</sub>		1.4
Rate of rise of solvent		7 fpm
Area of 1½ in IPS, Sch. 40 pipe		0.014 sq ft
Volume flow ratios	Scrub	1
	Feed	2
	Solvent	6
Distance from interface to solvent overflow		2 ft
Aqueous feed point to interface		11.25 ft
Aqueous discharge point to aqueous feed point		13.6 ft

The maximum solvent rate is calculated as follows:

$$\begin{aligned}
 \text{Solvent flow rate} &= (\text{Flooding rate, gal/hr/sq ft})(\text{cc/gal}) \\
 & \quad (\text{hr/min})(\text{area of col., sq ft})(\% \text{ of} \\
 & \quad \text{flooding/100})(\text{Solv. rate/total flow}) \\
 &= (500)(3,785)(1/60)(0.014)(50/100)(6/9) \\
 &= \underline{149 \text{ cc/min}}
 \end{aligned}$$

$$\begin{aligned}
 \text{(a) Solvent above} \\
 \text{the interface} &= (2)(0.78) \\
 &= \underline{1.56 \text{ ft of water}}
 \end{aligned}$$

The equivalent head of the solvent flowing through the column is calculated by multiplying the ratio of the solvent volume in the aqueous phase to the volume of the fluid space

Column Dimensions and Pressure Pot Locations (cont.)

available by the height of the fluid space. The solvent in the column is found by dividing the height of the aqueous phase containing solvent by the rate of rise of the solvent, and then multiplying the number of minutes thus obtained by the solvent flow rate.

$$\begin{aligned} \text{Vol. of solvent} & & & \\ \text{in aqueous} & = & (24/7)(149)(3.53 \times 10^{-5}) \\ & = & \underline{0.018 \text{ cu ft}} \end{aligned}$$

$$\begin{aligned} \text{Vol. of fluid} & & & \\ \text{space available} & = & (0.014)(24)(0.75) \\ & = & \underline{0.254 \text{ cu ft}} \end{aligned}$$

$$\begin{aligned} \text{(b) Equivalent head} & & & \\ \text{of solvent} & = & \frac{(0.018)(24)(0.78)}{0.254} \\ & = & \underline{1.70} \text{ ft of solvent or } \underline{1.33} \text{ ft of} \\ & & & \text{water} \end{aligned}$$

$$\begin{aligned} \text{(c) Scrub section,} & & & \\ \text{aqueous head} & = & \underline{[11.25 - (11.25/24.9)(1.70)]} \\ & = & \underline{11.5 \text{ ft of water}} \end{aligned}$$

$$\begin{aligned} \text{(d) Extraction section,} & & & \\ \text{aqueous head} & = & \underline{[13.6 - (13.6/24.9)(1.70)] \frac{2(1.4)+1.1}{3}} \\ & = & \underline{17.8 \text{ ft of water}} \end{aligned}$$

$$\begin{aligned} \text{(e) Aqueous between} & & & \\ \text{solv. fd. pt. \& } & & & \\ \text{aq. discharge} & = & (0.88)(1.3) \\ & = & \underline{1.1 \text{ ft of water}} \end{aligned}$$

Column Dimensions and Pressure Pot Locations (cont.)

(f) Aqueous head on discharge line = (x)(1.3) ft of water

(g) Air pressure on pressure pot = 0

f = a / b / c / d / e

1.3 x = 1.56 / 1.33 / 11.5 / 17.8 / 1.1

x = 33.3/1.3

= 25.6 ft

Allowing a margin of safety the pressure pot inlet was located at 20 ft - 6 in. above the floor or 19 ft-4 in. above the aqueous discharge nozzle.

When the column is being operated at the other extreme as at start-up, the liquid heads consist of the following:

- (a) Scrub section containing scrub solution
- (b) Extraction section containing feed solution
- (c) Aqueous discharge line to pressure pot containing scrub
- (d) Air pressure is being applied to pressure pot

Then a / b = c / d

(a) Scrub section, aqueous head = (11.25)(1.1)  
= 12.4 ft of water

(b) Extraction section, aqueous head = (13.6)(1.4)  
= 19.0 ft of water

Column Dimensions and Pressure Pot Locations (cont.)

(c) Aqueous hd. in discharge line = (19.3)(1.1)  
= 21.2 ft of water

(d) Air pressure on pressure pot = (x)(2.3) where x is psi of air

Again  $d \neq c = a \neq b$

$2.3 x \neq 21.2 = 12.4 \neq 19.0$

$2.3 x = 10.2$

$x = \underline{4.43 \text{ psi of air required}}$

A Foxboro Model 40 Stabilog flow controller planned for use on the column is normally operated at 17 psi maximum controlled pressure. A 3 to 1 Moore Pressure Reducing Relay will cut this pressure down to 5.67 psi, which exceeds that required as shown by the above calculations.

The overall length of the strip column was chosen as 19 ft 4 in., with the bottom of the lower spool piece 1 ft above the floor. The inlet of the pressure pot for the strip column was set at 15 ft. Both of these dimensions were determined by calculations similar to those described for the extraction column.

### 3.3 Evaporator

It is assumed that 141.3 gal of product solution containing 70 g/l of uranium will be evaporated to 2 M or 476 g/l of uranium. This requires a volume reduction factor of 0.147 resulting in a final volume of 20.8 gal with 120.5 gal being evaporated. If this were evaporated in a total of 40 hours, the maximum allowable time, the rate would have to be 3 gph or more. A surplus tantalum-lined evaporator with a nominal capacity of 10 gph of water has been located in Y-12 surplus. Since the evaporator is available at no cost other than the labor necessary to dismantle and transport it to X-10, and the capacity is in the correct range, its use seems desirable even though it has a greater capacity than is actually needed.

## 4.0 Solvent Extraction Equipment

### 4.1 Nomenclature

For purposes of brevity each equipment piece has been given a letter and number symbol. An example is "FI-XB-2", the symbol for the Aqueous Feed Rotameter. "FI" signifies **flow indicator** as given in the instrument code legend on drawing TD-1273, "X" differentiates the TBP Pilot Plant from other installations in building 706-HB, "B" designates the cell and the "2" is an arbitrary number. "M" indicates that the equipment is located outside of a cell. Pump symbols have a "P" following the numeral

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Nomenclature (cont.)

and agitator symbols are followed by an "AG". Steam jet syphons and air exhausters have been named by placing a "J" before the tank on the suction and discharge side of the jet. For example, JXC4-XM3 is a steam jet syphon which transfers liquid from tank XC4 to tank XM3.

4.2 Tanks and Columns

All of the major tanks, except XM-1 and XB-2, have been obtained from surplus in 9207-BT, Y-12. Tanks XM-1 and XB-2 had been previously used in the Redox installation in Bldg. 706-A Semi-Works. Vendors prints for all tanks obtained from 9207-BT are available in the Process Design group.

XB-1, feed preparation tank, has a capacity of 150 gallons, is constructed of Type 347 stainless steel and has an AEC-ORNL property number X-2407. It is equipped with the following nozzles:

- a. 6 in. IPS on which is mounted a Process Equipment Engineers, nozzle mounting, top-entering, direct-drive mixer with a 1/2 hp explosion-proof motor.
- b. 1/2 in. IPS -- thermocouple well.
- c. 3 in. IPS -- liquid level and specific gravity dip legs.
- d. 3 in. IPS -- dip leg to XB-2 and funnel inlet.
- f. 1 in. IPS -- jet suction to hot drain.

Tanks and Columns (cont.)

- g. 3 in. IPS -- fill from feed preparation area, and vent line.
- h. 1 in. IPS -- coil inlet.
- i. 1 in. IPS -- coil outlet.

By means of an air exhauster, JXB2-V, liquid from XB-1 is transferred to XB-2, the IAF feed tank. After proper valve manipulation a maximum of 50 psig air is applied to this tank forcing the aqueous feed solution to the extraction column. XB-2 is a 50 gal tank which, as previously mentioned, had been used as a feed tank in the Redox Semi-Works.

Tank XB-1, IBX or strip feed, is practically identical with XB-2. It is equipped with a calibrated sight glass which may be used to check the strip feed rotameter. It has a demineralized water inlet line, a vent line leading to the nitric acid system vent header, and a low pressure air inlet line.

XM-2, IAS or scrub feed, is a 30 gal tank constructed of Type 347 stainless steel, property number X-1702. It is equipped with the following nozzles:

- a. 1 in. IPS -- vent and low pressure air inlet.
- b. 1 in. IPS -- nitric acid inlet.
- c. 3/4 in. IPS -- connection for top of calibrated sight glass.
- d. 3/4 in. IPS -- blank.
- e. 1 in. IPS -- bottom outlet.

Tanks and Columns (cont.)

XB-2, IAX solvent feed tank, is another 150 gal. capacity vessel constructed from Type 347 stainless steel with a property number X-2449. It is equipped with the following nozzles:

- a. 6 in. IPS--blank.
- b. 1-1/2 in. IPS -- inlet from XC-8P solvent pump.
- c. 3 in. IPS -- low pressure air inlet.
- d. 3 in. IPS -- blank.
- e. 1-1/2 in. IPS -- vent.
- f. 1 in. IPS -- dip leg to solvent rotameter.
- g. 3 in. IPS -- top connection for calibrated sight glass.
- h. 1 in. IPS -- bottom outlet.

Tank XB-5, accumulator, is located in the solvent feed line to column XB-3. This tank is 6 in. in diameter by 18 in. long and has a capacity of approximately 490 cu in. It has 3/8 in. IPS connections on each end. The capacity of the accumulator is approximately equal to that portion of the column above the level at which the accumulator was installed. Its purpose is to provide a collection reservoir for the contents of the column in the event a break occurs in the solvent feed line outside the cell. Without such a vessel a leak may allow the contents of the column to syphon onto the floor outside of the shielded area.

Tanks and Columns (cont.)

Tanks XB-6 and XB-7 are pressure pots located in the aqueous discharge line from each column. Their purpose is to receive controlled air and apply it to the column in order that the organic-aqueous interface will be held at the proper level. Each pressure pot is constructed of an 18 in. length of 3 in. IPS Type 347 stainless steel pipe with a 1/4 in. IPS air inlet at the top, a 1/2 in. IPS liquid inlet 3-1/2 in. above the bottom, and a 1/2 in. IPS liquid discharge line on the bottom.

Tank XB-8, IAW Aqueous waste metering tank, is used to check the aqueous raffinate flow rate from the extraction column. It is constructed from a 48 in. length of 2 in. IPS, Type 347, stainless steel pipe. It is fitted with three 1/2 in. IPS flanged nozzles, an inlet at the top, an outlet at the bottom, and an overflow 2 in. from the top. The dip legs are 1/4 in. IPS pipe, one extending to the tank bottom and the other 1 ft above the tank bottom. The instrumentation is arranged in such a way that the interval necessary for the fluid to rise from the lower dip leg to the upper dip leg may be timed. The specific gravity may also be measured when the ends of both dip legs are immersed in the liquid. At a flowrate of 90 cc per min 7-1/2 minutes are required to contact both dip leg ends and 30 minutes are required to fill the tank. In the event the discharge valve is left closed after the reading is made, the liquid will merely overflow, bypassing the bottom outlet valve, and the run will not be hindered.

Tanks and Columns (cont.)

Tank XB-9 IAW aqueous waste tank, is constructed from Type 347 stainless steel, has a capacity of 75 gal and a property number X-1090. It is fitted with the following nozzles:

- a. 1 in. IPS -- blank.
- b. 1 in. IPS --vent.
- c. 3/4 in. IPS -- fill line from tank XB-8.
- d. 3/4 in. IPS -- blank.
- e. 1 in. IPS --bottom outlet to hot drain.
- f. 3 in. IPS -- liquid level and specific gravity dip legs.

The solvent raffinate flows from the strip column into tank XC-3, IBW waste solvent catch tank. This tank is also constructed of Type 347 stainless steel, has a capacity of 150 gal, and its property number is X-1294 . It is equipped with the following nozzles:

- a. 6 in. IPS -- blank.
- b. 1-1/2 in. IPS blank.
- c. 3 in. IPS-- liquid level and specific gravity dip legs.
- d. 3 in. IPS -- blank.
- e. 1-1/2 in. IPS -- waste solvent (IBW) inlet from strip column.
- f. 1 in. IPS -- vent.
- g. 3 in. IPS -- blank.
- h. 1-1/2 in. IPS -- bottom discharge to XC-1.

Tanks and Columns (cont.)

Upon completion of a run the solvent in XC-3 may be drained down to XC-1, solvent treatment tank. Here it may be given any type of aqueous wash desired. This tank's capacity is 75 gal, property number X-985, and it is constructed of Type 347 stainless steel. It is equipped with an agitator, XC-9 Ag, identical with agitator XC-10 Ag on XB-1, except that the stuffing box packing is suitable for contact with organic solvents. This tank has been altered in accordance with drawing TD-1267. In addition to the 6 in. IPS agitator nozzle, "a", this tank is equipped with the following nozzles:

- b. 1 in. IPS -- vent.
- c. 1 in. IPS -- bypass line from pump.
- d. 1 in. IPS -- fill line from XC-3, and funnel line.
- e. 1 in. IPS -- bottom drain line to solvent pump XC-8P.
- f. & g. 3/4 in. IPS -- sight glass.

The outlet line from XC-1 contains a section of 1 in. Pyrex pipe for use as a sight glass when separating the solvent- aqueous layers. Aqueous material may be discharged to the hot drain through a tee off the main line. Another connection is provided to allow fresh solvent to be introduced into the system from a drum.



Tanks and Columns (cont.)

XC-8P, XC-1 to XC-2 Pump, transfers solvent to solvent storage, back to XC-1, or to the solvent feed tank, XC-2. It is a Durco centrifugal pump, Series H5MBR60, property number X-2506, and is equipped with a 1/2 hp, 1725 rpm, 440 v explosion-proof motor. The wet end parts are constructed of Type 316 stainless steel. Previous to this installation it had been used in the 706-A Semi-works and had been originally obtained from 9207-BT surplus.

The aqueous stream bearing the uranium product collects in tank XC-4, IBP product catch tank. This tank was formerly WC-1 in the UAP Pilot Plant and was not moved from its former location. It is equipped with an agitator, XC-10 Ag, identical with XB-10 Ag. Following is the description of the tank nozzles:

- a. 6 in. IPS -- agitator.
- b. 3/4 in. IPS -- thermocouple well.
- c. 3 in. IPS -- liquid level and specific gravity dip legs.
- d. 3 in. IPS -- suction lines for jets JXC4-XM3 and JXC4-HD.
- e. 1/2 in. IPS -- aqueous product (IBW) fill line.
- f. 1-1/2 in. IPS -- sampler dip leg.
- g. 1 in. IPS -- vent.
- h. 1 in. IPS -- fill fine from funnel
- i. 3 in. IPS -- side outlet, blank.
- j. 1-1/2 in. IPS -- bottom outlet, blank.

Tanks and Columns (cont.)

This tank is also equipped with a 1 in. IPS low pressure steam coil which may be used, although its use is not contemplated.

The extraction and scrub column and the strip column, XB-3 and XB-4 respectively, are very similar to those used in Cell 2 in the 706-A Semi-works during "25" and Redox experiments. The assembly and details of these columns are shown on Dwg. TD-1254, Column XB-3 is constructed of 1-1/2 in. IPS Sch. 40, Type 347, stainless steel pipe. It is built in five sections, consisting of the lower spool piece, the packed extraction section, the aqueous feed spool piece, the scrub section, and the head piece which includes a side arm section of 1 in. Pyrex pipe for viewing the interface position. Discharge nozzles are 1/2 in. IPS while all feed nozzles are 3/8 in. IPS. All aqueous bearing nozzles discharge upwards and the solvent inlet nozzle points downward. A 1 in. diameter inverted cup is located just above the aqueous feed point for taking samples of solvent, and a thermocouple well is located two feet below the aqueous feed point. The side arm sight glass consists of a 1 in. length of Pyrex pipe. It is planned to view the interface through a hole in the cell wall by means of mirrors. A bubbler tube for use with the interface controller is located near the bottom of the head piece. Both sections are packed with 1/4 in. x 3/8 in.

Tanks and Columns (cont.)

stainless steel Raschig rings. A cone shaped packing support is located at the lower end of both the extraction and scrub sections.

The stripping column, XB-4, was originally planned to be constructed of 1-1/2 in. IPS, Sch. 40 pipe (1.610 in. ID). However, it was later increased to 2-1/2 in. IPS, Sch. 40 (2.47 in. ID) when the strip column flooding rate was found to be 500 gal/hr/sq ft compared to 1000 gal/hr/sq ft for the extraction column. The strip column uranium loss goes up rapidly as the number of strip stages is reduced, so the diameter of the strip column was proportioned to the extraction column diameter, permitting both columns to operate at the optimum flow rate for maximum stage efficiency. Column XB-4 consists of four sections -- the lower solvent feed-aqueous discharge spool piece, two sections of pipe packed with 1/4 in. x 3/8 in. stainless steel Raschig rings, and the top spool piece identical with that on column XB-3 except for the pipe diameter. Perforated conical packing supports are located at the lower end of each packed section, and thermocouple wells are located near the center of each section of pipe. The feed and discharge nozzles are of the same size and located in a manner similar to those on column XB-3.

4.3 Instrumentation

4.31 Liquid Level and Specific Gravity Indicators

All tanks inside the cells, except XB-2, XC-2, &XC-1, are equipped with liquid level and specific gravity manometers. XB-2 is a combination pressure and vacuum tank. Manometers have not been installed on this tank because of past difficulties with blowbacks in the manometer lines. XC-2, a solvent tank, is located inside the cell only for purposes of fire prevention. Outside the cell is located a sight glass which is connected to the tank through plug holes. XC-1 is located near the bottom of the cool cell and its sight glass may be viewed through the open cell door.

The system of remote liquid level and specific gravity indication is that commonly used in pilot plant installations at ORNL. A metered stream of air is bubbled through a dip line in the process vessel, and the pressure necessary to force the air out of the dip line is indicated on a manometer on the panel board. In most cases a separate dip line and air metering device is used for each high and low pressure manometer connection. Merriam Model A-203 stainless steel well-type manometers are used in all cases. All specific gravity manometers are 12 in. long.

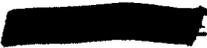
Liquid Level and Specific Gravity Indicators (cont.)

The liquid level manometers on tanks XC-3 and XC-4 are 30 in. long and those on tanks XB-9, XB-1 and XB-8 are 36 in. in length.

The manometers for XC-3 and XC-4, formerly used with the UAP Pilot Plant, had been equipped with Moore Constant Differential Relays for maintaining a constant air bubble rate with one setting of the air metering valve when liquid level changes occurred. This refinement was thought unnecessary and the differential relays were not installed when extra manometers were added for the TBP process. The lower ends of the specific gravity dip lines are separated by a vertical distance of 8 in. in all cases except the metering tank, XB-8, in which the distance is 12 in.

4.32 Column Feed Metering

Air pressure is used to force all feed liquids from pressure tanks to rotameters and thence to the column feed point. Taylor 1/4 in. air reducing valves, or equivalent, are used to maintain 10 psi on feed tanks XB-2, XC-2, and XM-1. A pressure relief valve set at 15 psi is located in each of these air lines as a safety measure. XB-2, IAF aqueous feed tank, requires 40 psi with a pressure release valve set at 50 psi.



Column Feed Metering (cont.)

Fischer and Porter Fig. 735 "Flowrators" with stainless steel floats and end connections are used as the primary flow-rate measuring devices. Table I shows the tube number and capacity of the four rotameters used.

Table I

Feed Stream	IAF FI-XB2	IAS FI-XM2	IAX FI-XC2	IBX FI-XM1
Tube No.	01-15	02-	01-15	01-15
Max. tube reading (cc/min. of H <sub>2</sub> O)	250	74	250	250

Feed tanks XM-2, XC-2, and XM-1, are equipped with calibrated sight glasses for checking rotameter calibrations. The upper sight glass connection is made directly to the tank while the lower end is connected to the feed line with a valve between the connection point and the feed tank. Thus, when a check is desired, the valve can be closed, and the liquid will flow under tank pressure from the calibrated sight glass. Sampling valves are provided on the discharge side of each rotameter which allows liquid to be collected in a graduate and the flow

Column Feed Metering (cont.)

rate determined, barring such difficulties as runback from the feed line between the rotameter and column. The only method of checking FI-XB2, IAF rotameter, is to determine the aqueous flow rate by using metering tank XB-8 and subtracting the scrub (IAS) feed rate.

4.33 Interface Controllers

The method of controlling the column interfaces is similar to that used in recent ORNL Semi-works and Pilot Plant column installations. A recording differential pressure controller is provided to sense the deviation of the interface position by noting the change of mass of liquid above an air inlet point near the top of the column. The instrument then applies the necessary air pressure to the pressure pot which causes the interface position to rise or fall as may be required. A jack leg is provided on the discharge side of the pressure pot to prevent the applied air pressure from merely blowing out into the discharge line. The instruments required to control each interface are as follows: A Fisher and Porter C-Clamp Flowrator for metering the air to the column head piece, a Foxboro Stabilog Model 40 flow controller, and a 3-1 Moore pressure reducing relay for

Interface Controllers (cont.)

the extraction column. The controlled air output of this controller is 0 to 17 psi. and the 3 to 1 pressure reducing relay is used to bring the controlled pressure down to a usable range of 0 to 5.67 psi. A 5-1 pressure reducing relay is used on the strip column resulting in a controlled pressure range of 0 to 3.4 psi.

4.34 Temperature Recording Instrument

A 6-point Brown Electronik recording potentiometer is provided to measure the temperature of tank XB-1, the discharge water from XM-5, the evaporator condenser, two points on XB-4, the strip column, and one point on the extraction column XC-4. The temperature sensitive elements are iron-constantan thermocouples.

4.35 Miscellaneous Instruments

Ammeters with a range of from 0 to 5 amperes are provided for remotely operated agitators, XB-10 Ag and XC-10 Ag, on tanks XB-1 and XC-4, respectively. These ammeters indicate whether or not the agitators are operating properly. Pressure gages with a range of 0 to 160 psi with 2 psi sub-divisions are used on the steam lines to steam syphons and heating coils. Numerous other small pressure gages are used as required.

#### 4.4 Sampling

The samplers are of two types, viz , tank samplers in which material is sucked from the process vessel, and stream samplers in which the liquid is obtained by opening a valve in a process line. A list of all the sample points is given in the Appendix. The tank samplers for XB-1 and XC-4 are connected to the sampling station previously used for the UAP Pilot Plant installation. In this installation a Schutte & Koerting, Fig. 217 Syphon is used as an air exhauster to pull liquid from the tank being sampled into a 1 in. x 12 in. section of Pyrex pipe. By proper manipulation of valves, low pressure air is then applied to the Pyrex pipe, and the liquid is blown back to the tank thus rinsing the sampler line. After repeating this sequence of operations several times, another valve is opened at the bottom of the Pyrex pipe and a sample is taken.

Stream samplers have been constructed by bringing the process line involved out of the cell to the sampling station and inserting a tee and valve in the line. Thus, the sample can be obtained by merely opening the valve and allowing the liquid to run into the sample container.



5.0 Product Evaporator Equipment

5.1 Tanks

From XC-4, IBP product catch tank, the dilute product is transferred by steam syphon to tank XM-3, evaporator feed tank. This tank is made of Type 347 stainless steel, has a capacity of 150 gal, and a property number of X-2291. The nozzles in use are a 1 in. IPS, fill from XC-4, a 1-1/2 in. IPS vent, a 3 in. IPS top sight glass connection, a 1-1/2 in. IPS bottom outlet. All other nozzles are blanked off.

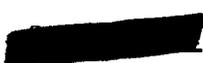
Tank XM-6, condensate catch tank, is a 75 gal, Type 347 stainless steel tank whose property number is X-1163. It has a 1 in. IPS nozzle which serves as the condensate fill point, a 1 in. IPS vent line, a sight glass, and a 1 in. IPS bottom discharge line.

Tank XM-8, concentrate cooler, is a 14 in. length of jacketed 2 in. IPS pipe with both inlet and outlet connections swaged down to 1 in. IPS. connections. The water inlet and outlet jacket connections are 3/4 in. IPS. XM-8 is installed in such a way that it maintains the correct liquid level in the evaporator as well as serving as a concentrate cooler.

Tank XM-9, concentrate catch tank, may be any convenient vessel for collecting the concentrated uranium solution. A 55 gal stainless steel drum will probably be used.

## 5.2 Evaporator and Condenser

As previously mentioned, the evaporator and condenser have been obtained as a unit from Y-12 surplus. The evaporator consists of a vertical steel tube lined with tantalum which was constructed by the Fansteel Metallurgical Corp. The boiler portion of the evaporator is 7 in. in diameter with an effective length of 18 in. The maximum allowable steam pressure is 150 psi gage. The steam inlet is a 1-1/4 in., 150 lb. standard flanged nozzle, and the steam discharge nozzle is a 3/4 in., 150 lb standard flanged pipe. The upper or de-entrainment portion of the evaporator is 10 in. inside diameter by approximately 4-1/2 ft high. It had been originally packed with 36 in. of Fiberglass for de-entrainment purposes, but it is not planned to use such packing in this operation unless absolutely necessary. The top flange of the evaporator is equipped with a 3/4 in. and a 1 in. flanged nozzle, both of which are blanked off. Vapor leaves the evaporator through a 3 in. flanged connection on the side near the top. The concentrate is removed through a 1 in. nozzle close to the bottom of the evaporator. The vapor leaves the evaporator and enters a 6 in. x 6 in. x 3 in. tantalum-lined tee which is mounted on top of the condenser. The condenser is also constructed of steel lined with tantalum and tapers from 6 in. ID down to 2 in. ID and is 60 in. long. A full length water jacket equipped with 1-1/4 in. IPS flanged inlets and 1-1/2 in. IPS flanged outlets, surrounds the condenser.



5.3 Instrumentation

The evaporator has a bare minimum of instrumentation as automatic operation is not desired. Feed to the evaporator is metered by a Fischer and Porter Fig. 735 "Flowrator" with a 2A-25A tube and a maximum calibration reading of 1600 cc. per min. of water. After leaving the concentrate cooler the concentrated product may either flow upward through a 1-1/2 in. by 12 in. length of Pyrex pipe and thence out via the side outlet of 1-1/2 in. x 1-1/2 in x 1-1/2 in. Pyrex tee which contains a hydrometer and thermometer, or it may go directly to the concentrate catch tank, XM-9. In order to prevent collapsing the tantalum liner of the condenser, the process water line to the condenser is equipped with a pressure reducer, gage, and a 28 ft. open-end stand pipe. The steam line to the evaporator is equipped with a steam pressure reducer and gage.

6.0 Costs

Table II gives the actual costs incurred on this project.

Table II

Engineering	\$4,664
Labor	8,492
Materials	<u>12,765</u>
Actual Cost	\$25,921

No charge is included for the surplus equipment obtained from 9207-BT, Y-12, except labor for dismantling and transportation. Overhead is also not included in the actual cost figure.

7.0 Appendix

7.1 Equipment Lists

7.11 Tanks, Columns, Agitators, and Pumps

XM-1	IBX strip feed
XM-2	IAS scrub feed
XM-3	Evaporator feed tank
XM-4	Evaporator
XM-5	Condenser
XM-6	Condensate catch tank
XM-7	(No equipment corresponds to this number)
XM-8	Concentrate cooler
XM-9	Concentrate catch tank
XB-1	Feed adjustment tank
XB-2	IAF feed tank
XB-3	Extraction and scrub column
XB-4	Strip column
XB-5	Accumulator
XB-6	Pressure pot for XB-3
XB-7	Pressure pot for XB-4
XB-8	Aqueous Waste metering tank
XB-9	IAW aqueous waste tank
XB-10 Ag	Agitator for XB-1
XC-1	Solvent treatment tank

Tanks, Columns, Agitators, and Pumps (cont.)

XC-2 IAX solvent feed tank  
XC-3 IBW Waste solvent catch tank  
XC-4 IBP product catch tank  
XC-8P XC-1 to XC-2 pump  
XC-9Ag Agitator on XC-1  
XC-10Ag Agitator on XC-4

7.12 Steam Jet Syphons

The steam jet syphons are all Schutte & Koerting Fig. 217 syphons fabricated from Type 316 stainless steel. They have a 1 in. IPS screwed inlet and discharge and a 3/4 in. IPS screwed steam inlet. The numbers of the jets are:

JXB1-HD  
JXB9-HD  
JXC4-XM3  
JXC4-HD  
JXC2-XC-1

7.13 Exhauster

The one exhauster used in the system is a Schutte & Koerting Fig. 431 air jet exhauster with threaded connections. The suction and discharge connections are 3/4 in. and the air inlet is 3/8 in. IPS. The code number given the exhauster is JXB2-V.

7.14 Tank Samplers

- ST-1 XB-1 sampler
- ST-2 XC-4 sampler

7.15 Stream Samplers

- SS-1 IAF Between FI-XB-2 and XB-3
- SS-2 IAX Between FI-XC-2 and XB-5
- SS-3 Organic at aqueous feed point
- SS-4 IAS Between FI-XM-2 and XB-3
- SS-5 IAW Between XB-6 and XB-8
- SS-6 IAP Between XB-3 and XB-4
- SS-7 IBP Between XB-7 and XC-4
- SS-8 Condensate Between XM-5 and XM-6
- SS-9 Solvent Between XC-8P and XC-1
- SS-10 IBW Between XB-4 and XC-3
- SS-11 Evap. feed. Between XM-3 and XM-4
- SS-12 Concentrate Between XM-8 and XM-9
- SS-13 Concentrate Between XM-6 and CD.

7.2 Drawing List

- TD-1254 Column XB-3 and XB-4 -- Assembly and Details
- TD-1255 Tank XB-8 -- Assembly
- TD-1256 Tank XB-7 -- Assembly
- TD-1266 Tank XB-5 -- Assembly
- TD-1267 XC-1 -- Tank Alterations

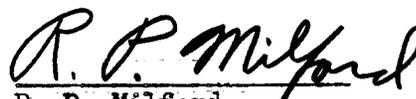
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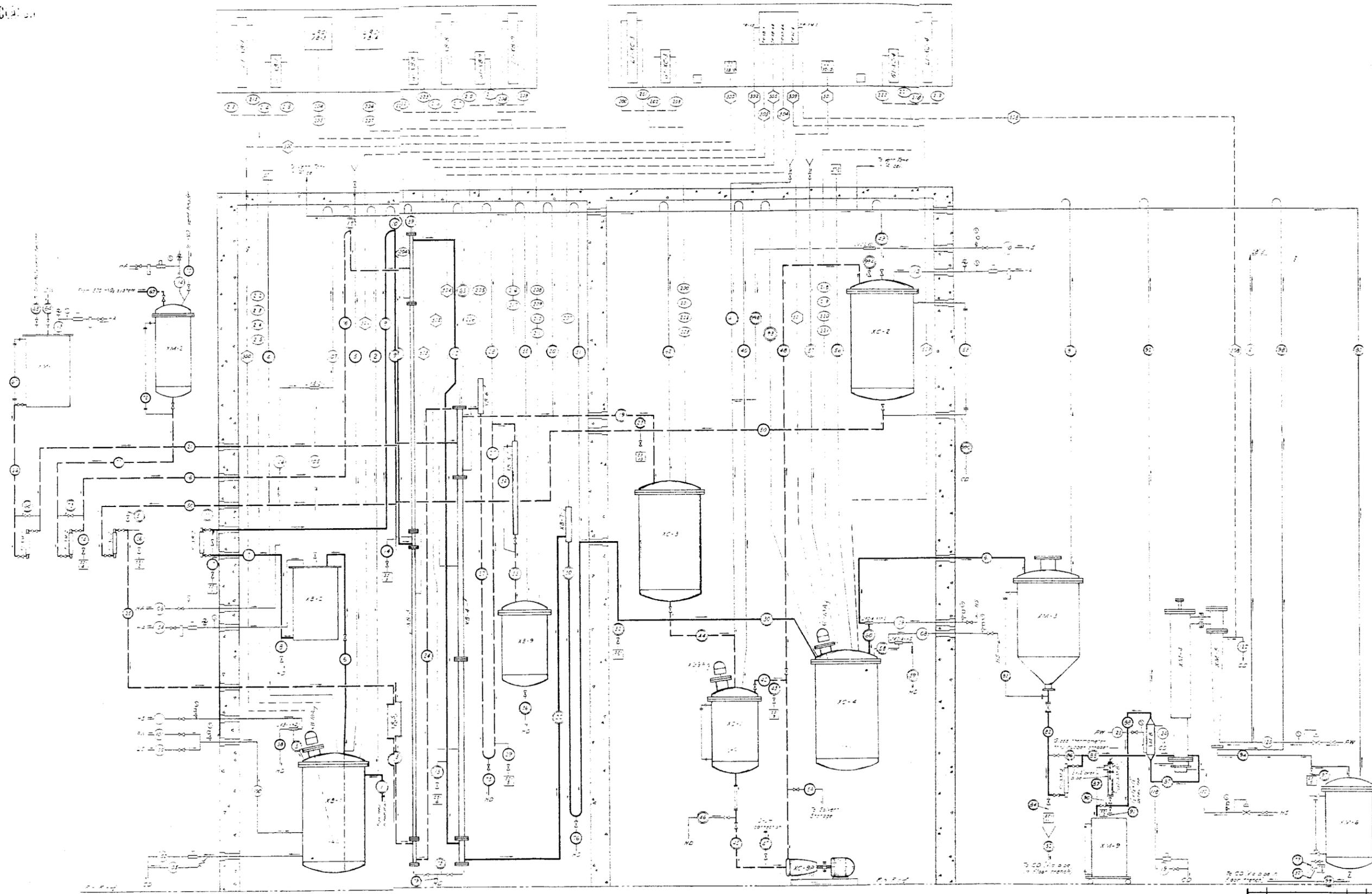
- TD-1270 Equipment Location
- TD-1271 Panel Board and Rotameters -- Locations and Details
- TD-1273 Equipment Flowsheet
- TD-1285 Tank Support -- Structure and Details (for evaporator & condenser)
- TD-1317 Schematic Flowsheet and Pipe Schedule
- SK-37 Installation Details for XM-4 and XM-5

8.0 Acknowledgment

The contribution of all who had a part in this installation is gratefully acknowledged. Design information as determined by laboratory personnel under the direction of T. C. Runion and D. E. Ferguson was obtained from Mr. Ferguson. E. O. Nurmi contributed much to the design and provided the engineering supervision from the Technical Division during construction. R. V. Foltz did the bulk of the drafting and routine engineering, with Harry Watts and his group providing the balance of the drafting work.

D. E. Ferguson, E. O. Nurmi, and F. L. Culler lent valuable assistance in the preparation and review of this report.

  
R. P. Milford  
Design Engineer



**LEGEND**

**INSTRUMENT CODE**

LI.....Liquid level indicator  
 GI.....Specific gravity indicator  
 TR.....Temperature recorder  
 II.....Current indicator  
 FI.....Flow indicator  
 LRC.....Level recorder & controller

**SERVICE CODE**

HS.....High pressure steam  
 LS.....Low pressure steam  
 HA.....High pressure air  
 LA.....Low pressure air  
 FW.....Filtered water  
 PW.....Process water  
 HD.....Hot drain  
 CD.....Cold drain  
 DW.....Demineralized water

**SYMBOLS**

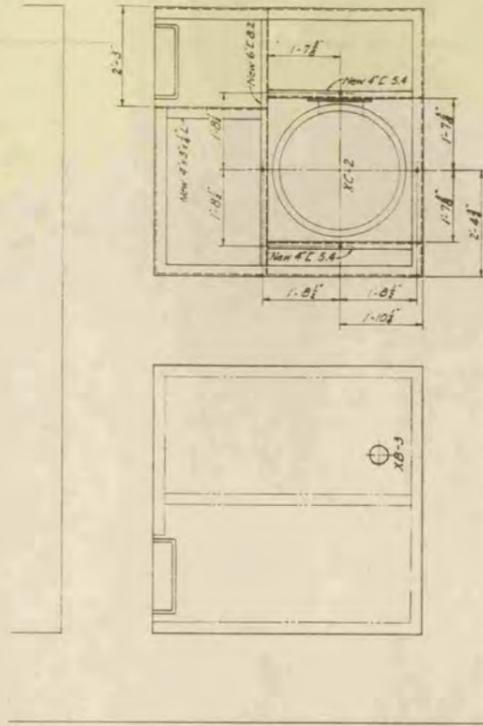
○.....Process piping  
 ○.....Service piping  
 ○.....Electrical lines  
 ○.....Instrument lines  
 □.....Sample unit  
 Y.....Funnel  
 ——— Jet  
 ——— Trap

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 R P M/1-5

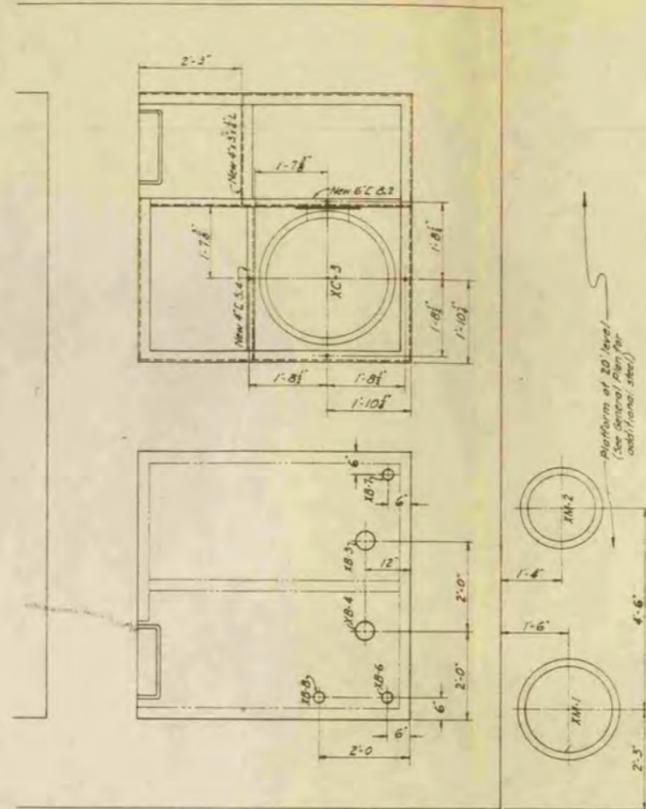
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TECHNICAL DIV.	P.O. BOX P	OAK RIDGE, TENN.
BLDG 706-118		
TBP SEMI-WORKS		
EQUIPMENT FLOWSHEET		
DRAWN BY	DATE	SCALE
CHECKED BY	DATE	

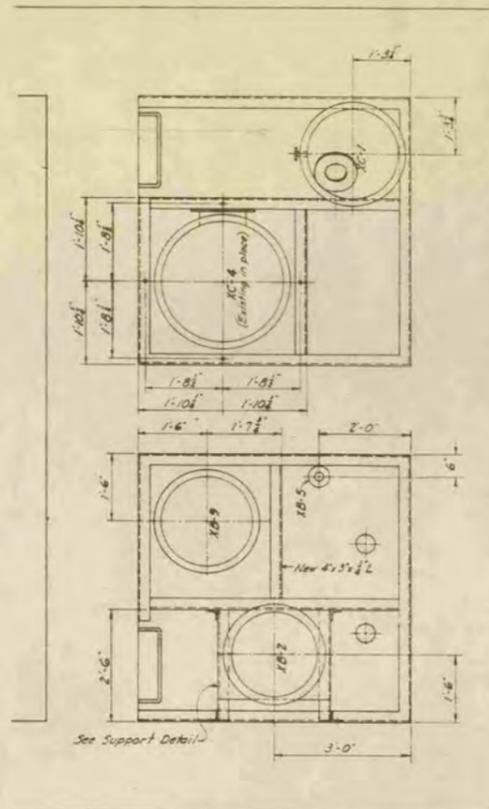
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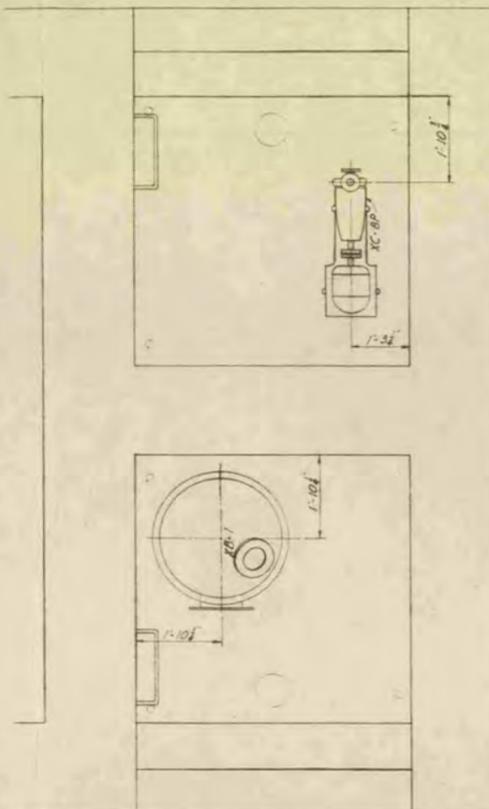
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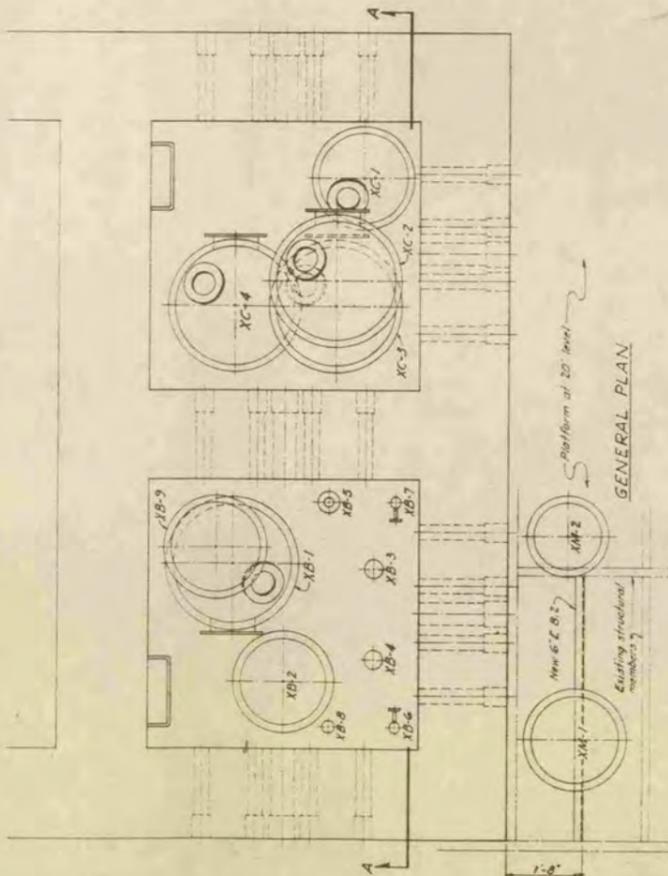
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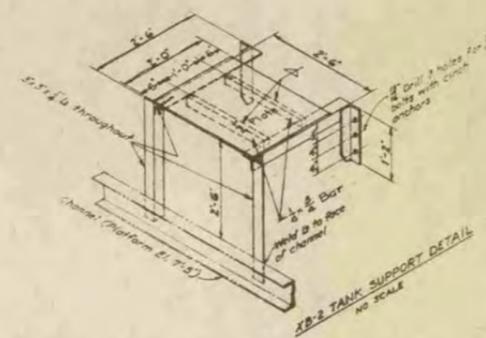
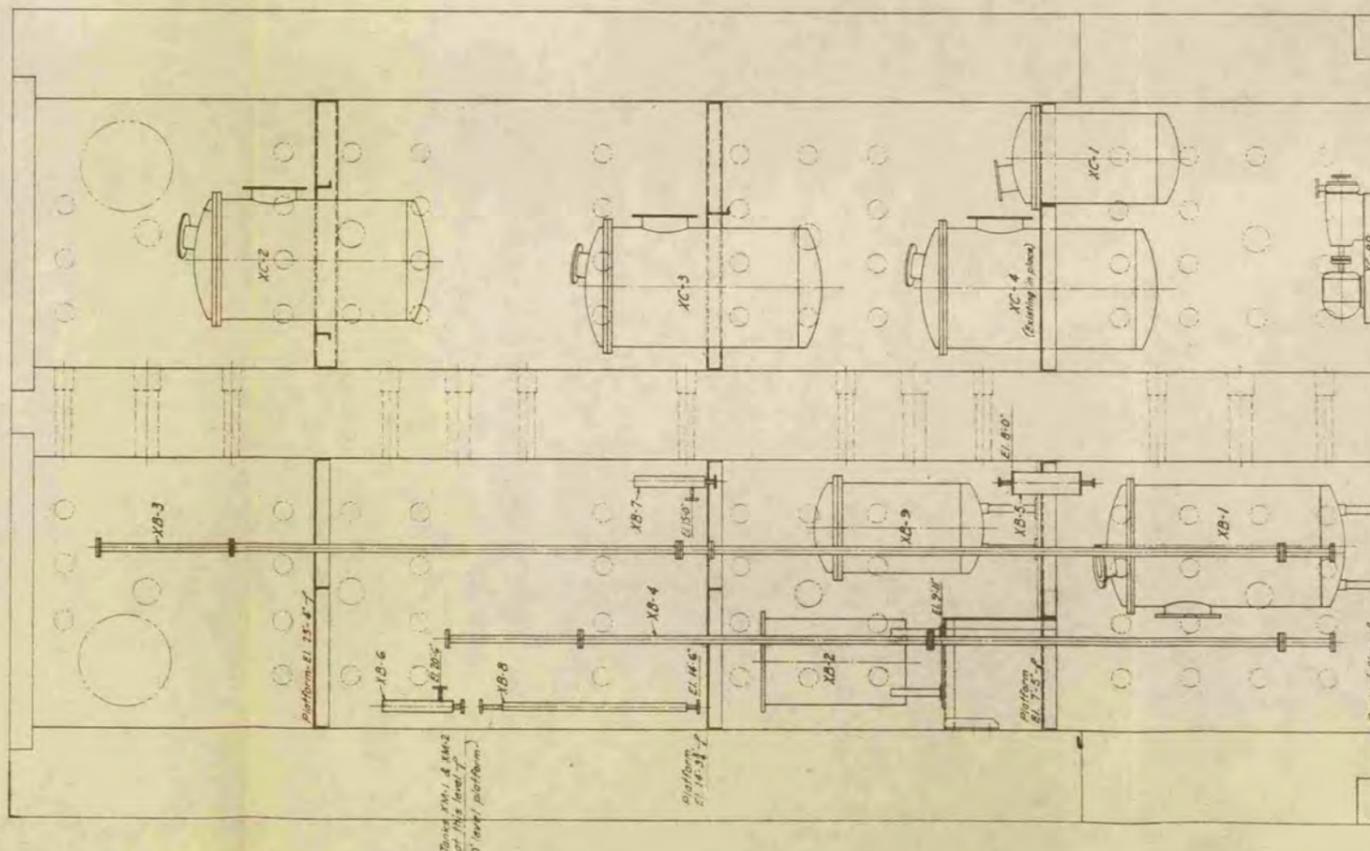
PLAN - PLATFORM EL. 7'-5"



PLAN - GROUND FLOOR



GENERAL PLAN

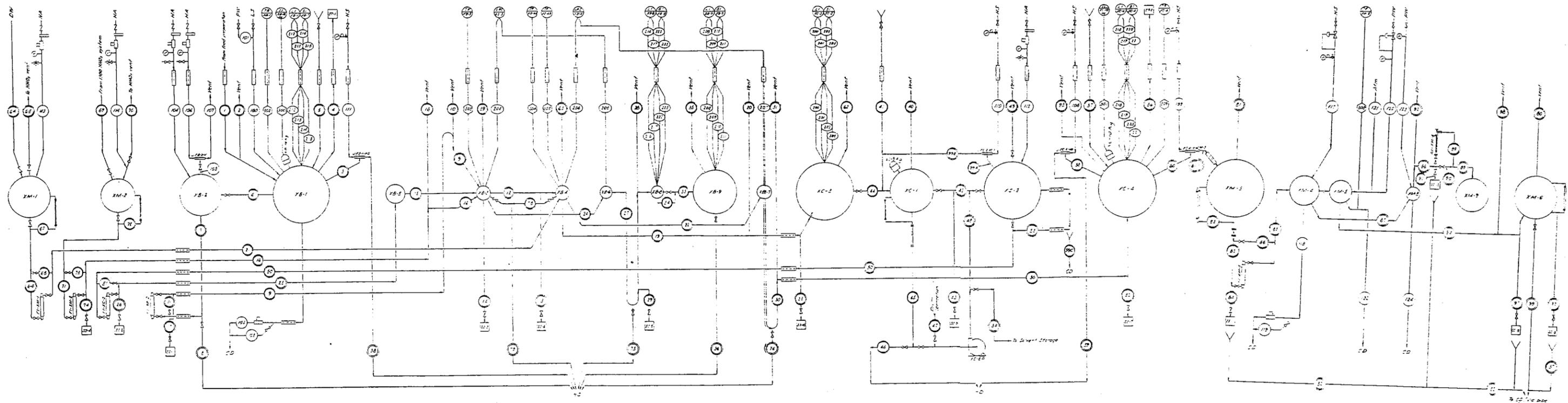


GENERAL NOTES:  
 1- All welded construction.  
 2- For anchoring equipment drill 1/2" holes for 3/8" mach bolts.  
 3- Painting of new steel shall conform with paint of existing steel as directed by the Job Engineer.

NOT CLASSIFIED

Dwg. 8129

DO NOT SCALE THIS DRAWING		OAK RIDGE NATIONAL LABORATORY	
APPD. DATE	REV. NO.	TECHNICAL DIV.	PO. BOX #
		BLDG. 706-HB	OAK RIDGE, TENN.
TBP SEMI-WORKS			
EQUIPMENT LOCATION			
DESIGNED BY	DATE	SCALE	1/2" = 1'-0"
CHECKED BY	DATE		



PROCESS LINES				SERVICE LINES (cont'd.)				SERVICE LINES (cont'd.)			
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100	1	FM	100-100	200	1	FM	100-100	200	1	FM	100-100

**INSTRUMENT CODE**

LI.....Liquid level indicator  
 SI.....Specific gravity indicator  
 TR.....Temperature recorder  
 NI.....Current indicator  
 FI.....Flow indicator  
 LRC.....Level recorder controller

**SERVICE CODE**

HS.....High pressure steam  
 LS.....Low pressure steam  
 HA.....High pressure air  
 LA.....Low pressure air  
 PW.....Process water  
 HD.....Hot drain  
 CD.....Cold drain  
 DW.....Demineralized water

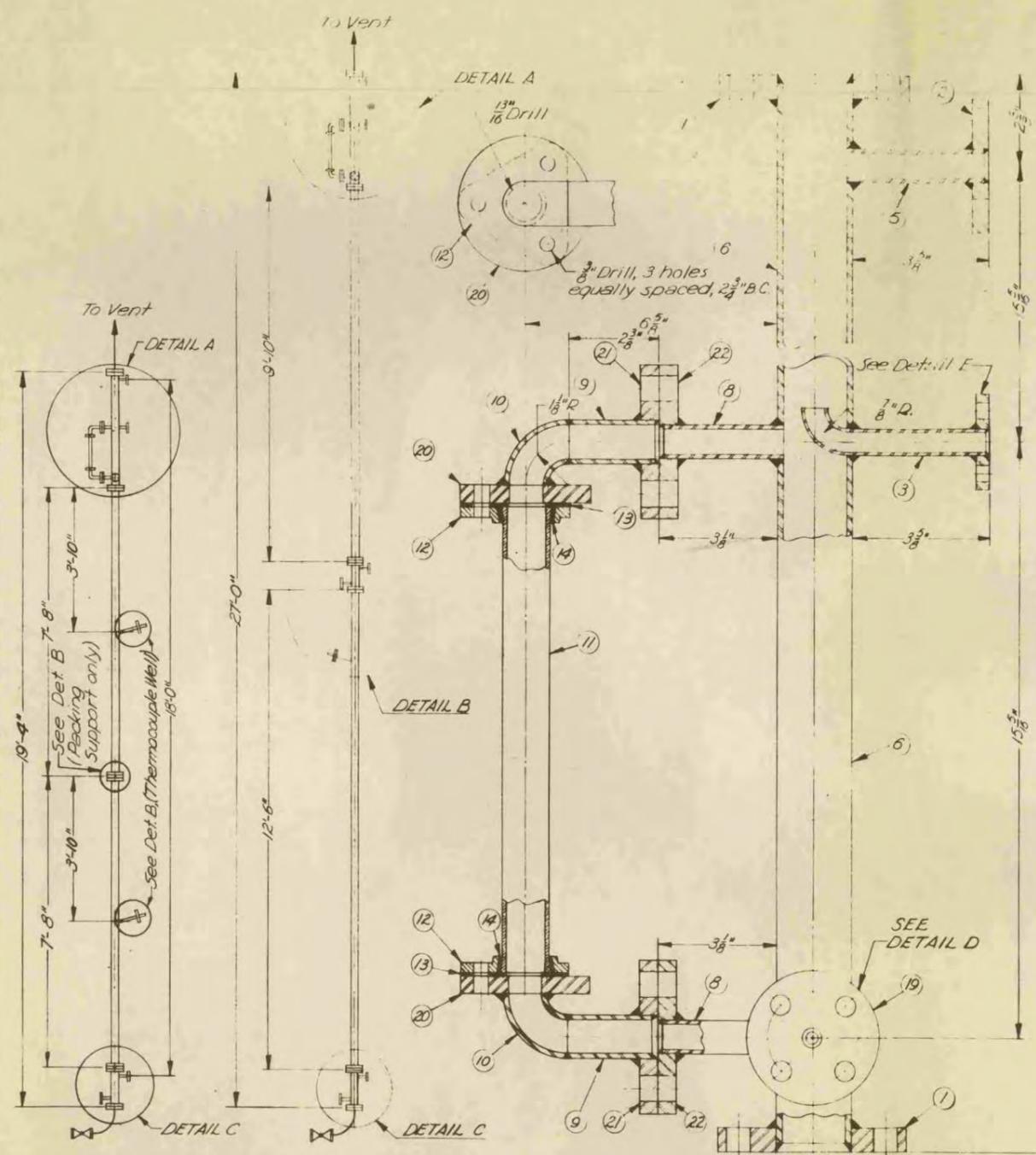
**SYMBOL LEGEND**

○.....Process piping 0-99  
 ○.....Service piping 100-199  
 ○.....Electrical lines 200-299  
 ○.....Instrument lines 300-399  
 □.....Sample unit  
 Y.....Funnel  
 ⊢.....Plug  
 ⊢.....Jet  
 ⊢.....Trap

Dwg. 8130

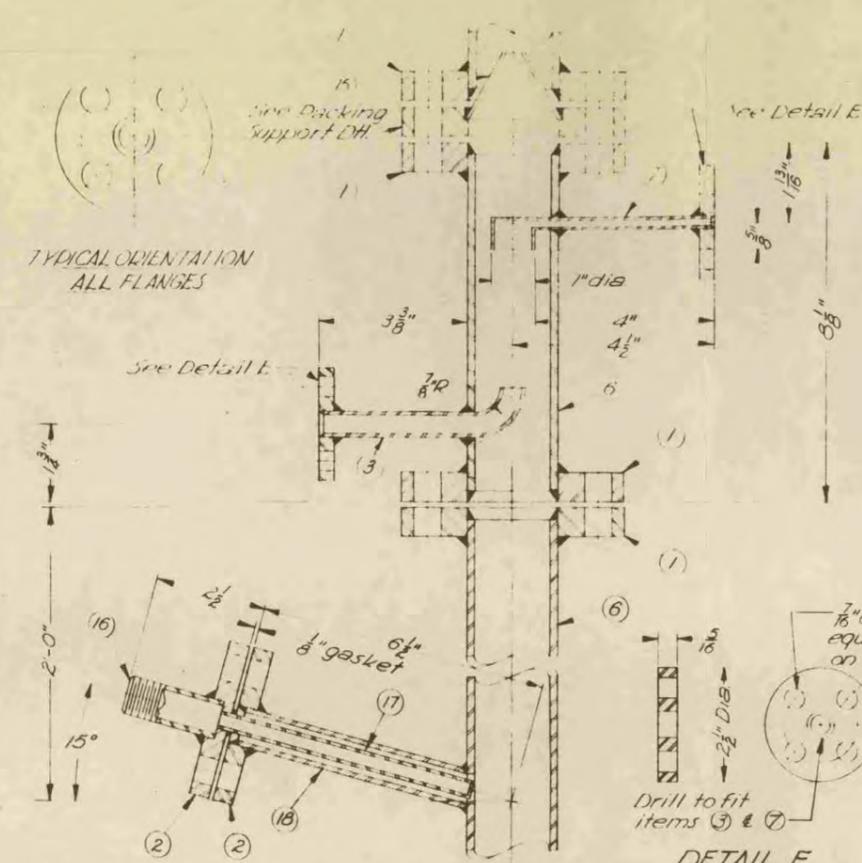
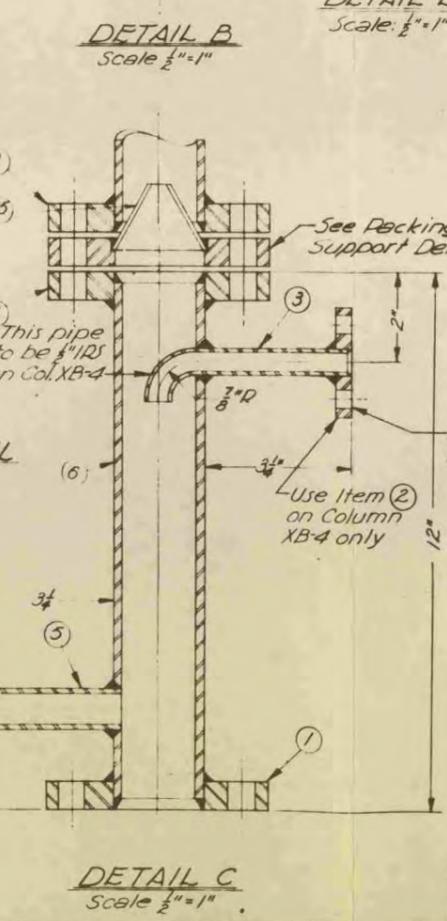
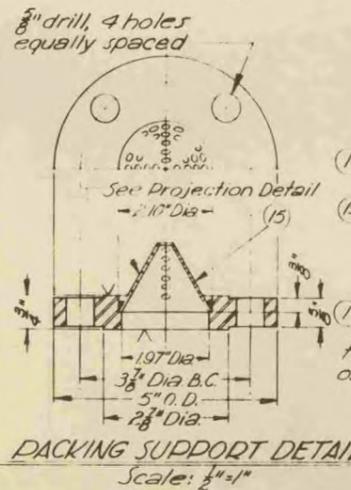
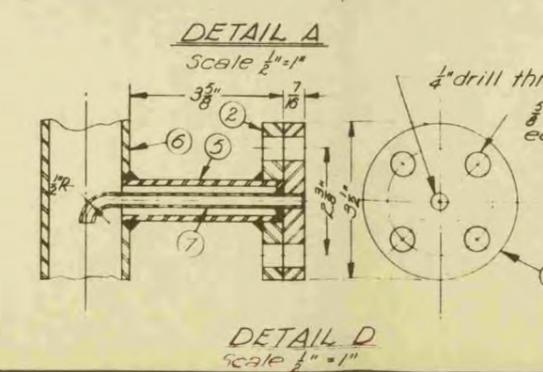
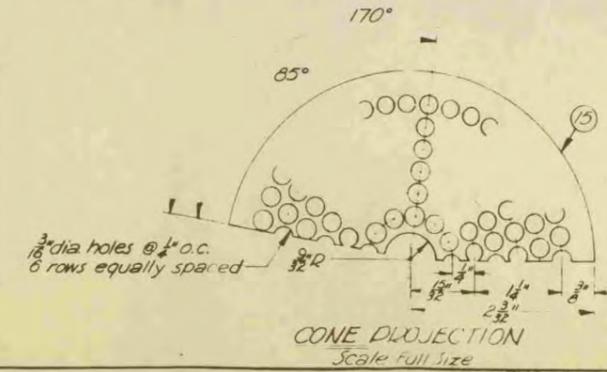
NOT CLASSIFIED

APPRO. DATE	DAK RIDGE NATIONAL LABORATORY
TECHNICAL OFF.	PO. BOX #
BLDG.	106-NB
TBP	SEMI-WORKS
SCHEMATIC FLOWSHEET	
DESIGNED BY	DATE
DATE	SCALE
DATE	SCALE



ASSEMBLY  
Scale 1/2" = 1'-0"  
COLUMN XB-4

ASSEMBLY  
Scale 1/2" = 1'-0"  
COLUMN XB-3



ITEM NO.	QTY	DESCRIPTION	MATERIAL	REMARKS
1	18	1/2" IPS Std. Slip-on Weld Flange	S.S.	
2	13	1/2" IPS Std. Slip-on Weld Flange	S.S.	
3	4	3/4" IPS Pipe	S.S.	
4	4	3/4" IPS Pipe	S.S.	
5	6	3/4" IPS Pipe	S.S.	
6	16	1/2" IPS Pipe (Sch. 40)	S.S.	
7	1	3/4" Tubing	S.S.	
8	4	3/4" IPS Pipe	S.S.	
9	4	3/4" IPS Pipe	S.S.	
10	4	3/4" IPS 90° welding elbow	S.S.	Std. weight-long radius
11	2	1" x 12" Pyrex pipe	Glass	
12	4	1" Corning Flange	C.I.	Corning Glass Works
13	4	1" Full face gasket 8" thick	Polythene	
14	4	1" Corning Insert	C.I.	Corning Glass Works
15	4	16 Ga. 062 (U.S.S.) Sheet	S.S.	
16	3	3/4" IPS Pipe	S.S.	
17	3	3/4" IPS (Sch. 40 pipe)	S.S.	
18	3	3/4" IPS Pipe	S.S.	
19	2	See Detail D		See Detail D
20	4	7/8" Plate	S.S.	Drilled for Items 10 & 12
21	4	3/4" IPS Std. Slip-on Weld Flange	S.S.	
22	4	3/4" IPS Std. Blind Flange	S.S.	Drilled for Items 8 & 21

Above Bill of Material for both Columns (XB-3 & XB-4)

NOTES:

1. All material shall be Stainless Steel Type 304 except where otherwise noted.
2. All welds shall be free from loosely deposited metal and free from pits and cracks.
3. Corning Glass Works cast iron flanges shall receive two coats of acid resistant paint.
4. All flanges to be 150lb Std. slip-on welding unless otherwise specified.
5. Use Lincoln Stain weld A5-Cb, A7-Cb, Arcos Chromend, or Stainlend 19/9 Cb welding rods.
6. Use 3/8" sheet Polythene gaskets and stainless steel studs and nuts where required.

NOT CLASSIFIED

FOR REPORTS ONLY

Fig. IV  
Dwg. 8131

APPD. DATE		OAK RIDGE NATIONAL LABORATORY	
TRU 7-20-49		TECHNICAL DIV. — P.O. BOX P — OAK RIDGE, TENN.	
7064B BLDG.			
TRD SEMI-WORKS			
COLUMNS XB-3 & XB-4			
ASSEMBLY & DETAILS			
DRAWN BY	DATE	SCALE AS NOTED	
C.M. DEW	7-12-49		
CHECKED BY	DATE	DRAWING NO.	REV.
R.P.M.	7-1-1950	FD-1254	
REV. NO.	REVISION	APPD. DATE	